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# AN EVALUATION OF METAL SALTS OF NTO AS POTENTIAL PRIMARY EXPLOSIVES

MRL-TN-563

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# An Evaluation of Metal Salts of 3-Nitro-1,2,4-triazol-5-one (NTO) as Potential Primary Explosives

Lance D. Redman and Robert J. Spear

MRL Technical Note MRL-TN-563

# **Abstract**

Sodium, potassium and ammonium salts of NTO were prepared, and used as intermediates to prepare a range of heavy metal salts; silver, lead, mercuric, barium, cadmium, strontium, cupric, nickel and stannous. Normal salts were prepared in all cases, and acid salts for sodium, potassium, lead and mercuric. The ammonium salt was shown to be identical to that reported previously. Response to mechanical and thermal stimuli is typical of sensitive secondary explosives. It is concluded that these materials do not have any potential for use as primary (initiating) explosives.

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# An Evaluation of Metal Salts of 3-Nitro-1,2,4-triazol-5-one (NTO) as Potential Primary Explosives

## 1. Introduction

The fillings in most initiating devices and systems which contain primary explosives are based on only three types of materials:

- 1. Lead azide in detonators.
- 2. Lead or barium styphnates in primers, igniters and delays, and
- 3. Tetrazene, to sensitise mechanically initiated systems.

All have safety or operational performance problems, and there has been considerable R & D effort expended over the years to identify materials capable of replacing them [1]. Despite this effort, which has been carried out throughout the Western defence community, very few new materials have met the stringent safety and performance requirements necessary and been subsequently introduced into service systems. The only new primary explosives which have been introduced to other than very specialised applications are mercuric 5-nitrotetrazole in the US [2] and silver azide in the UK [1].

As part of our technology base R & D on Insensitive Munitions, we have carried out a preliminary assessment of 3-nitro-1,2,4-triazol-5-one (NTO) as an insensitive high explosive filler in main charge warheads and fuze boosters [3]. NTO is a relatively acidic molcule (pKa 3.67) [4] and should readily form a range of metallic salts of the type  $M^{n+}[NTO-H]^{-}$  or  $M^{n+}[NTO-2H]^{-1}$ , as shown below. Since NTO is an energetic material, it is expected that some of these salts could have the properties required of a primary explosive.

Manchot and Noll [5], who were first to prepare NTO in 1905, also prepared a silver salt which was characterised only by elemental analysis;  $C_2HN_4O_3Ag$ , i.e.  $Ag^+[NTO-H]^-$ . Chipen et al [6] prepared a potassium salt,  $K^+[N\Gamma O-H]^-$ . $H_2O$  and a mercuric salt,  $Hg^{2+}[NTO-2H]^{2-}$ , both being characterised only by elemental analysis; they also mentioned a water soluble lithium salt and sparingly soluble barium, sodium and silver salts. None of these salts were assessed for their explosive properties, nor were any explosive hazards described.

The only salts of NTO which have been considered for their explosive properties are the ammonium salt,  $NH_4^+(NTO-H)^-.H_2O$ , as well as the analogous salts derived from ethylenediamine aminoguanidine [4, 7]. None of these salts, nor those from similar non-energetic amines, are likely to show properties of primary explosives, and the principle potential use of such materials is in low melting eutectic explosives with ammonium nitrate [4].

We therefore set out to prepare a range of metal salts of NTO, and to screen them as potential primary explosives using a few simple tests. Any of these materials showing promise would then be properly characterised and assessed using a wider range of procedures. The results of this investigation are detailed in this report.

# 2. Experimental

#### 2.1 Materials

NTO, recrystallised from water, was available from our previous study [3]. All other chemicals and solvents were laboratory grade, water was distilled.

Microanalysis was performed by Chemical and Micro Analytical Services Pty Ltd, Victoria.

#### Potassium and Sodium Salts

#### (i) Acid Salts

A solution of potassium or sodium hydroxide (0.04 mol) in water (20 mL) was heated to 80°C then poured into a solution of NTO (0.04 mol) in water (20 mL) preheated to 80°C. The mixture was maintained at 80°C for 15-20 min then allowed to cool to ambient temperature over 2-3 h. The crystalline product was recovered by gravity filtration and allowed to dry in air.

#### (ii) Normal Salts

The method was exactly as described for the acid salts except that the mole ratio of hydroxide:NTO was increased to 2:1, i.e. 0.08 mole of hydroxide in 20 mL of water.

#### Ammonium Salt

To a solution of NTO (0.04 mol) in water (20 mL) at ambient temperature was added conc. ammonia solution (1 mL, sp. grav. 0.88 g/mL). The mixture was allowed to stand at room temperature, then the crystalline product was isolated by filtration and dried in air. Found: C, 14.5%; H, 4.3; N, 43.1.  $C_2H_7N_7O_4$  requires C, 14.6; H, 4.3; N, 42.4, corresponding to  $NH_4^+(NTO-H)^-.H_2O$  (see Section 3.2).

#### Barium, Strontium and Stannous Salts

#### (i) Acid Salts

A solution of NTO in aqueous sedium hydroxide (each 0.04 mol, total 40 mL) at 80°C was added, in one portion, to a solution of the corresponding chloride (0.02 mol) in water (20 mL) preheated to 80°C. The mixture was held at this temperature for 15-20 min, then was allowed to cool slowly to ambient temperature over 2-3 h. The product was isolated by filtration and allowed to dry at ambient temperature.

#### (ii) Normal Salts

The procedure was exactly the same as the acid salt except the mole ratios were 1:1, i.e. a solution of the corresponding chloride (0.04 mol) in water (20 mL) was added.

#### Lead Salts

The procedures differed in two ways from the barium etc. salts described above: the reaction was carried out at 50°C, because there was some evidence of decomposition at 80°C, and lead nitrate (rather than chloride) was employed.

#### Silver Salt

A solution of NTO (0.04 mol) and conc. ammonia (1 mL) in water (20 mL) at 80°C was treated in one portion with a solution of silver nitrate (0.08 mol) in water (20 mL) preheated to 80°C. The mix was reacted at 80°C for 15-20 min then allowed to cool to ambient temperature over 2-3 h. The crystalline product was isolated by filtration and allowed to dry at ambient temperature.

#### Mercuric Salts

The methods used were exactly as described for the silver salt. The acid salt was prepared using mercuric nitrate (0.02 mol) in water (20 mL), and the normal salt was prepared using 0.04 mole in 20 mL of water.

# Cadmium, Cupric and Nickel Salts

The method was exactly as described for the mercuric salts except that the chlorides were used. Cally a normal salt was prepared in each case, i.e. 0.04 mole of the chloride in 20 mL of water was the reagent employed.

# 2.2 Preliminary Assessment of Hazard

A small amount of the salts were hit with a hammer on a hardened steel anvil, and burnt on a piece of paper.

#### 2.3 Thermal Response

Hot Stage Microscopy

Thermal behaviour was initially studied at a heating rate of '0°C/min using a Leitz Ortholux Optical Microscope with an attached Mettler FP-2 hot-stage.

Differential Scanning Calorimetry (DSC)

DSC thermograms were recorded using a Perkin-Elmer 1980-20. Differential Scanning Calorimeter equipped with a Perkin-Elmer DS 3600 Data Station. All samples were examined in open aluminium sample pans under a flowing nitrogen atmosphere at a heating rate of 10°C/inin. Temperatures were corrected (lead) and heats of reaction were determined directly from peak areas.

Temperature of Ignition [8a]

Temperature of Ignition (T of I) was determined using an instrument built to specification for the ERDET of I test. A sample of 50 mg in a glass test tube was heated at 5°C/min till ignition occurred, defined by either visible signals such as smoke/frame or audible hiss/bang. The T of I is the temperature at which this event occurs; usually duplicate samples were tested.

#### 2.4 Mechanical Response

Ball and Disc [8b], Rotter Impact [8c]

Sensitivity to impact was initially assessed by the Ball and Disc method using an instrument constructed to ERDE specifications [8b]. No ignitions were observed at the maximum drop height of 30 cm for any of the materials examined.

The only material tested for Rotter impact was the silver salt. This sample had relatively low bulk density and only 25-26 mg could be weighed into each brass cap. The caps were placed overnight in a desiccator, then fitted in turn over a polished sized anvil and impacted by a 5 kg weight falling from a preset height. Go/no-go was delineated by > 1 ml gas evolution from the sample for a positive result. Impact heights were varied in a typical Bruceton procedure with a total of 20 caps being tested.

The resulting Figure of Insensitiveness (F of I) is quoted relative to RDX grade G = 80 and is rounded to the nearest 5 units. Gas evolution represents the average for all positive results.

#### 3. Results and Discussion

# 3.1 Preparation of NTO Salts

Initial experiments were carried out to prepare the sodium, potassium, magnesium and ammonium salts; not being heavy metal salts, it was felt that these would present the lowest hazards to handling.

The potassium and sodium salts were readily prepared by neutralisation of NTO using aqueous hydroxide solution, and the ammonium salt using concentrated aqueous ammonia. The potassium and ammonium salts crystallised readily from solution, while the sodium salts could not be induced to crystallise even after substantial evaporation of water. The sodium salts were subsequently obtained by addition of alcohol to the aqueous solutions. The magnesium salt could not be induced to form a suitable product, probably due to its highly hydrated nature, and was not further pursued.

Heavy metal salts were prepared firstly on microscope slides by allowing a single crystal of the metal salt, usually the chloride or nitrate, to react with each of three solutions:

- (a) a 0.2 M aqueous solution of NTO
- (b) an aqueous solution of NTO neutralised with potassium hydroxide
- (c) an aqueous NTO solution neutralised with excess ammonia

It was usually found that (a) resulted in a poorly defined gelatinous product, while (b) and/or (c) gave a crystalline material with acceptable handling properties.

This screening process showed that the barium, strontium, stannous and lead salts could best be prepared via the sodium salt, chosen over the potassium salt because of greater solubility, while the silver, mercuric, cadmium, cupric and nickel salts were prepared via the ammonium salt. All reactions were performed at 80°C, except for the lead salt where there had been some evidence of decomposition during the experiments on microscope slides at this temperature; 50°C was the chosen reaction temperature in this case.

A scheme showing the formation of the "normal salts", i.e. those in which all acidic hydrogens on the NTO have been neutralised, is given below. The corresponding "acid salts"  $M^{n+}[NTO-H]_n$  were prepared using half the sodium hydroxide with half the metal salt, except the mercuric salts where the mercuric: NTO mole ratio was halved. Ordy normal salts were prepared from cadmium, cupric and nickel ions.

Scheme 1 Preparation of normal salts of NTO from divalent heavy metal ions (hydration not shown)

#### 3.2 Molecular Formulae/Structure of the NTO Salts

The salts prepared in 3.1 above were in general not characterised for molecular formulae or structure. This was a deliberate policy; the principal aim was to screen materials for their potential as primary explosives. Any with promising properties would have been subsequently examined in considerably more detail.

The sodium and potassium salts were found to produce different crystalline forms when the ratio of hydroxide to NTO was 2:1 or 1:1. It was assumed that the former were normal salts, as shown above in Scheme 1, and the latter were acid salts. The lead and mercuric normal and acid salts were also noticeably different. The physical form, as well as an assessment of flow/handling properties, of all the salts prepared is listed in Table 1.

Also listed in Table 1 are proposed molecular formulae for the salts (cf. structures in Introduction). These formulae were derived from published data for the potassium [6], mercuric [6] and silver [5] salts coupled with differential scanning calorimetry data (DSC, see Table 2). In particular, the DSC thermograms permitted estimation of the degree of hydration since water molecules of crystallisation could be detected by their endothermic absorptions in the 100-140°C range due to "evaporation" from the crystals. Anhydrous materials such as the silver and lead salts could therefore be identified since they lacked such endotherms. The nickel salt was quite hydrated with at least two water molecules per metal atom. More precise definition of hydration could have been obtained from DTG via mass loss associated with these endotherms, but this was unavailable when this project was carried out. It should be noted that the DSC thermograms for the normal sodium and potassium salts both contain two endotherms, while the decomposition exotherms are similar to the acid salts. This

could mean that the normal salts are dihydrates, or could alternatively mean that the "normal" salts, as isolated, are mixtures of normal and acid salts. No attempt was made to resolve this.

The molecular formula of the ammonium salt was investigated because an ammonium salt had been extensively characterised previously [4] and shown to have the structure ANTO shown below. We noted that whereas the reported melting point of ANTO was 190°C [4], the material we isolated had a markedly different behaviour when examined by hot stage microscopy; there was partial "melting" over the temperature range 136-141°C, followed by a small amount of "melting" in the 185-195°C range. At the completion of these two stages, the product consisted of crystals which subsequently underwent decomposition above 260°C. This is totally consistent with the DSC thermogram (Table 2); we attribute the first "melting" to dehydration.

The differing behaviour to that reported for ANTO [4] led us to the hypothesis that we had isolated a bis-ammonium salt (shown below). However, microanalytical data conclusively showed that the salt we had isolated was ANTO: found C, 14.5%; H, 4.3 and N, 43.1. ANTO requires C, 14.6%; H, 4.3 and N, 42.4. In contrast the bis-ammonium salts require C, 13.2 or 12.0 (x = 1 or 2); H, 5.5 or 6.0 and N, 46.2 or 42.0. The "melting point" reported in [4] is clearly a simplification of the thermal response.

## 3.3 Explosive and Hazard Properties

Initial screening tests (hammer, burn on paper) indicated minimal hazard but were discouraging with respect to explosive properties. All materials responded only marginally to hammer blow, ignition being accompanied by the occasional puff of smoke but no noise. When burned on filter paper the general response was fizzing or spluttering, although the lead salt gave a fairly rapid burn. These are properties typical of high explosives, not primary explosives.

These conclusions were reinforced by testing using ball and disc. This is the standard screening test for mechanical sensitiveness of primary explosives [8b] and all materials gave no response at maximum (30 cm) drop height. A full Rotter impact test [8c] was carried out on the silver salt, chosen as having the best handling properties of all the materials prepared. The Figure of Insensitiveness, 130 (RDX = 80), indicates relative insensitivity while the average gas evolution of 1.4 mL indicates poor ability to propagate from ignition.

Thermal responses as measured by the Temperature of Ignition test [8a] (50 mg heated at 5°C/min) are listed in Table 2. In general the materials responded by smoke or flame at 210-280°C, but the cadmium and silver salts gave explosions above 300°C while the lead and sodium salts gave explosions at lower temperatures.

The clear conclusion is that these materials behave like sensitive secondary explosives, and should be handled accordingly. They are unlikely to have any potential as primary explosives.

Energoutput is also fairly modest (Table 2), ranging from 1.289 kJ/g for the cadmium salt to 0.36 kJ/g for the acid lead salt. For comparison, the figures for normal and basic lead styphnate are 1.96 and 1.17 kJ/g respectively [9].

# 4. Conclusions

A range of alkali and heavy metal salts have been prepared from NTO. The heavy metal salts were prepared via the intermediacy of the sodium or ammonium salt, and in general had reasonable handling properties including flow.

Both acid and normal salts were prepared from sodium, potassium, lead and mercuric, and normal salts for the remainder. About half the salts were hydrated, the hydration being qualitatively determined using DSC. The strontium and nickel salts appeared to be too hydrated to be suitable for use as explosives.

The molecular formula of only one compound, the ammonium salt, was determined in detail. This was necessitated because the properties of the compound isolated here differed from those reported previously [4]. However, microanalytic data showed the compound to be the same as the reported structure ANTO.

Both impact and thermal sensitiveness data identify the NTO salts as having properties of sensitive secondary explosives, not primary explosives. Both ignition and propagation occur only with difficulty, and in most cases an explosion does not result. Heat output is only modest. Because of these unpromising properties, explosive performance was not investigated further.

# 5. Acknowledgements

The technical contributions of Mr Ewan Wanat and Mrs Branka Pletikapa, who performed T of I, Ball and Disc and Rotter Impact, and Mr Mark Fitzgerald, who performed DSC measurements, are very gratefully acknowledged.

Dr Dan Whelan is thanked for his contributions via a number of technical discussions.

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Table 1 Physical Description of Metal Salts of NTO Prepared at MRL

Salı	Suggested molecular Formula	Physical Form	Flow Properties	
Sodium, acid salt	Na(NTO-H).H <sub>2</sub> O <sup>8</sup>	Fine yellow needles	Poor	
Sodium, normal salt	Na <sub>2</sub> (NTO-2H).2H <sub>2</sub> Ob	Fine yellow-orange needles	Poor	
Potassium, acid salt	Na <sub>2</sub> (NTO—2H).2H <sub>2</sub> O <sup>b</sup> K(NTO—H).H <sub>2</sub> O <sup>c</sup>	Bright yellow crystals	Acceptable	
Potassium, normal salt	$K_2(NTO-2H)^2H_2O^b$	Orange-yellow crystals	Good	
Silver, acid salt	Ag(NTO-H)d	Fine orange crystals	Good	
Lead, acid salt	Pb(NTO-H)2	Orange powder	Acceptable	
Lead, normal salt	Pb(NTO-2H)	Bright yellow crystals	Good	
Mercuric, acid salt	$Hg(NTO-\cdot H)_2$	Yellow powder	Acceptable	
Mercuric, normal salt	Hg(NTO-2H) <sup>C</sup>	Orange powder	Good	
Barium, normal salt	$Ba(NTO-2H).H_2O^a$	Orange powder	Good	
Cadmium, normal salt	Cd (NTO-2H)	Orange powder	Good	
Strontium, normal salt	Sr(NTO-2H).2H <sub>2</sub> O <sup>e</sup>	Fine yellow needles	Poor	
Cupric, normal salt	Cu(NTO-2H)	Green powder	Good	
Nickel, normal salt	Ni(NTO2H).2H <sub>2</sub> O <sup>f</sup>	Fine, green-black crystals	Good	
Stannous, normal salt	Sn(NTO-2H)	Yellow crystals	Poor	
Ammonium	$NH_A(NTO-H).H_2O^g$	Fine yellow needles	Poor	

a DSC data (Table 2) indicates hydration is the same as the potassium acid salt which has been shown to be a monohydrate [6].

- c From ref [6].
- d From ref [5].
- e Several endotherms in DSC, at least 2H<sub>2</sub>O.
- f Large dehydration endotherm in DSC, at least  $2H_2O$ .
- g Structure proposed in ref [4], confirmed in this study by elemental analysis (ANTO).

b Possibly a dihydrate since two distinct endotherms in the DSC (Table 2). See discussion in Section 3.2.

Table 2 Thermal (T of I, DSC) and Mechanical (Ball and Disc, a Rotter F of Ib) Sensitiveness of NTO Metal Salts

Salt	T of I, 5°C/min (°C, duplicates)	DSC, 10°C/min Endotherms/exotherms (°C) Ex	otherm (kJ/g)
Na (acid)		broad endotherm $\sim 120\text{-}180$ sharp exotherm, onset $\sim 250$ , max 268.9	0.744
Na (normal)	238, 239 explodes	endotherms 90-175 2 or 3 exotherms	
K (acid)	242 smoke and noise	broad endotherm $\sim 130\text{-}165$ sharp exotherm, onset $\sim 250$ , max 260.4 broad exotherm, onset $\sim 290$ , max 307.9	0.686 0.310
		total	0.996
K (normal)	243, 244 popping noise	two or three endotherms 130-180 sharp exotherm, onset $\sim 234$ , max 259.3 less sharp exotherm, onset $\sim 283$ , max 313.9	0.511 0.5
		total	1.028
Ag	302, 302 explodes	small endotherm 130-170 two overlapping exotherms, onset ~ 284, max ~ 301, 311.3	0.756
Pb (normal)	212, 213 explodes	broad exotherm, onset $\sim 232$ , max 249.5 following weak exotherm, max 316.2	0.584 0.084
		total	0.668
Pb (acid)		broad exotherm, onset $\sim 218$ , max 263.4 very weak following exotherm, max $\sim 315$	0.360
		total	0.360
Hg (normal)	232, 234 yellow smoke	broad exotherm, onset ~ 231, max ~ 259	0.536
Hg (acid)		broad exotherm, onset $\sim 190$ , max $\sim 248$	0.459
Ba (normal)	230, 230	broad endotherm $\sim 100\text{-}155$ broad exotherm, onset $\sim 242$ , max 278.7	0.620
Cd (normal)	314, 316 explodes	broad exotherm, onset ~ 293, max 331.4	1.289
Sr (normal)	228, 230 popping noise	s eral endotherms 80-180°C	
Cu (normal)	237, 238 black smoke and noise	two overlapping exotherms, onset $\sim 218$ , max 264 (broad) and 301.4 (sharp)	0.902
Ni (normal)	280, 281 flames	very large endotherms 140-240 broad exotherm onset √ 311, max 316.2	0.589

Table 2 (continued)

Salt	T of I, 5°C/min (°C, duplicates)	DSC, 10°C/min Endotherms/exotherms (°C)	exotherm (kJ/g)
Sn (normal)	252, 256 yellow-orange smoke	exotherm onset ∿ 258, max 261.6	0.384
NH <sub>4</sub>	213, 214 yellow smoke	strong broad endotherm $\sim 100\text{-}140$ followe by second multipeaked endotherm $\sim 150\text{-}2$ sharp exotherm, onset $\sim 269$ , max 276.4	

a All samples gave 10/10 no fires at maximum (30 cm) drop height.

b Only one sample assessed; Ag (acid), F of I 130, average gas evolved 1.4 mL.

c Possibly indicates a mixture of the normal and acid salt.

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Sodium, potassium and ammonium salts of NTO were prepared, and used as intermediates to prepare a range of heavy metal salts; silver, lead, mercuric, barium, cadmium, strontium, cupric, nickel and stannous. Normal salts were prepared in all cases, and acid salts for sodium, potassium, lead and mercuric. The ammonium salt was shown to be identical to that reported previously. Response to mechanical and thermal stimuli is typical of sensitive secondary explosives. It is concluded that these materials do not have any potential for use as primary (initiating) explosives.